

SYSTEM AND PROCESS FOR THE OXYGEN DELIGNIFICATION OF PULP CONSISTING OF LIGNOCELLULOSE-CONTAINING MATERIAL

The present invention relates to a system and a  
5 process for oxygen delignification in accordance with  
the preambles to patent claim 1 and patent claim 9,  
respectively.

State of the art

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A number of different processes for oxygen  
delignification are known.

US,A,4.259.150 presents a system with multistage oxygen  
bleaching in which, in each stage, the pulp is first  
15 mixed to a lower consistency with O<sub>2</sub>, water and NaOH,  
followed by a thickening back to the consistency level  
which the pulp had prior to the stage in question. The  
aim is to obtain an economic, chlorine-free bleaching  
with high yield. At the same time, the kappa number can  
20 be lowered, by means of repeated stages, from 70 down  
to 15 or even less than 15.

SE,C,467.582 presents an improved system for  
the oxygen bleaching of pulp of medium consistency. By  
means of controlling the temperature in an optimized  
25 manner, an oxygen bleaching takes place in a first  
delignification zone at a low temperature, with this  
being followed by a second delignification zone at a  
temperature which is 20-40 degrees higher. The aim is  
to obtain an improved yield and an improved viscosity,  
30 while retaining the dwell time, in association with  
industrial use.

Other variants of oxygen delignification in two  
stages have also been patented in addition to  
SE,C,467.582. SE,C,505.147 presents a process in which  
35 the pulp should have a high pulp concentration in the  
range of 25-40% in the first stage and a concentration  
of 8-16% in the second stage, at the same time as the

- 2 -

temperature in the second stage should be higher than, or equal to, the temperature in the first stage, in line with the temperature difference which is recommended in SE,C,467.582. The advantages of the solution in accordance with SE,C,505.147 are stated to be the possibilities of admixing more oxygen in the first high-consistency stage without there being any risk of channel formation but where, at the same time, unused quantities of oxygen can be bled off after the first stage in order subsequently to be admixed in a second mixer prior to the second stage.

SE,C,505.141 presents a further process which is an attempt to circumvent SE,C,467.582, since that which it is sought to patent is stated to be that a temperature difference between the stages does not exceed 20 degrees, i.e. the lower suitable temperature difference patented in SE,C,467.582, but that a temperature difference should nevertheless be present. In addition to that, it is stated that a) the pressure should be higher in the first stage and b) that the dwell time is short in the first stage, i.e. in the order of magnitude of 10-30 minutes, and also c) the dwell time in the second stage is longer, i.e. in the order of magnitude of 45-180 minutes.

A lecture entitled "Two stage MC-oxygen delignification process and operating experience", which was given by Shinichiro Kondo from the Technical Div. Technical Dept. OJI PAPER CO. Ltd. At the 1992 Pan-Pacific Pulp & Paper Technology Conference ('99 PAN-PAC PPTC), Sept. 8-10, Sheraton Grande Tokyo Bay Hotel & Towers, presents a successful installation which was constructed with two-stage oxygen delignification in 1986 in a plant in Tomakomai. In this OJI PAPER plant in Tomakomai, the pulp was fed, with a pressure of 10 bar, to a first oxygen mixer (+ team) followed by an after-treatment in a "preretention tube" (prereactor), with a 10 minute

- 3 -

dwelt time in which the pulp pressure is reduced to a level of about 8-6 bar due to pipe losses, etc. After that, the pulp was fed to a second oxygen mixture followed by an after-treatment in a reactor at a pressure of 5-2 bar and with a dwell time of 60 minutes. It was stated at this point that preference would have been given to having a "preretention tube" which would have given a dwell time of 20 minutes but that it was not possible to construct this due to lack of space. OJI PAPER stated that, by using this installation, they had succeeded in obtaining an increase in kappa reduction at a lower cost in chemicals and with the pulp viscosity being improved.

Most of the the prior art has consequently been directed towards a higher pressure in the first reactor at a level of about 6(8)-10 bar. A pressure in the first reactor of up to 20 bar has even been discussed in certain extreme applications. This results in it being necessary to manufacture the reactor spaces which are required for the first delignification zone such that they can cope with these high pressure levels, with a consequent requirement for substantial material thickness and/or good material qualities, which in turn result in an expensive installation.

In pulp suspensions in industrial production processes, there are large quantities of readily oxidizable constituents/structures which already react under modest process conditions. It is therefore advantageous, in a first stage, to add oxygen in quantities which are such that this part of the pulp which is relatively easily oxidized is allowed to oxidize/react first of all. Severe problems arise if an attempt is made to compensate for this by overadding oxygen since there is the immediate danger of canalization problems (as mentioned in the said SE,C,505.147).

- 4 -

Object of the invention

One object of the invention is to avoid the disadvantages of the prior art and to obtain an oxygen delignification which gives increased selectivity. The invention permits an optical practical application of the theories regarding a first rapid phase and a second slower phase during the oxygen delignification process, with the optimal reaction conditions being different between the phases.

At the high hydroxide ion concentrations and high oxygen partial pressures which are conventionally employed in the first stage, the carbohydrates are attacked more than is necessary, thereby impairing the quality of the pulp. A lower oxygen partial pressure, and preferably a lower temperature as well, in the first stage as compared with the second stage decreases the rate of reaction for the breakdown of carbohydrates more than it decreases the rate of reaction for the delignification, leading in turn to an increase in the total selectivity on the pulp after the two stages.

Another object is to allow a simpler and cheaper process installation in which at least one pressure vessel, in a first delignification zone, can be manufactured using thinner material and/or using a lower material quality which is suitable for a lower pressure class.

Yet another object is also to make it possible to use steam at moderate pressure especially when there is a need to increase the temperature substantially between the first and second stage and when the pressure in the second stage is considerably higher than that in the first stage. In most cases, the supply of medium-pressure steam and low-pressure steam is very good in a pulp mill whereas high-pressure steam is in short supply due to the large number of processes which require high-pressure steam. This also makes it

- 5 -

possible to convert existing single-vessel delignification systems where, with the previously the prior art for converting to a two-stage design, a restriction has been imposed by the fact that the  
5 prevailing pressure in the plant's steam grid has not enabled a sufficiently large quantity of steam to be admixed with the pulp in order to achieve the desired temperature in the second delignification stage.

Yet another object is to optimize the mixing  
10 process in each position such that only that quantity of chemicals/oxygen is added which is consumed in the subsequent delignification zone and where the admixture of chemicals/oxygen does not need to compete with the simultaneous admixture of steam for the purpose of  
15 increasing the temperature to the desired level. In this way, it is possible to dispense with bleeding systems for overshooting quantities of oxygen at the same time as it is possible to reduce the total consumption of oxygen, which in turn reduces the  
20 operating costs for the operator of the fibre line and thus shortens the pay-off time. At the same time it is possible to select a smaller size of dynamic mixer for admixing chemicals, which mixer is dimensioned solely for the volumes of chemicals which are actually being  
25 admixed.

Yet another object is to increase, in an oxygen delignification system having a certain total volume of the first and second stages, a so-called H factor by operating the first stage for a short time at low  
30 temperature and operating the second stage for a longer time at a higher temperature. Thus, in connection, for example, with conversions of existing single-vessel oxygen delignification stages, a simple conversion, including a small prereactor and a modest increase in  
35 the reaction temperature in the existing reactor, can increase the H factor and at the same time improve the selectivity over the oxygen stages.

- 6 -

The invention is described in more detail with reference to the figures in accordance with the following figure list.

5 Figure list

Figure 1 shows a system for oxygen delignification in two stages in accordance with the invention; AND  
Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages which are  
10 gained relative to the prior art with regard to reduction in kappa number and an increased H factor.

Description of embodiment examples

15 Figure 1 shows an installation, according to the invention, of a system in an existing plant in which the oxygen delignification process needed upgrading.

An existing first MC pump 1 (MC = medium  
20 consistency, typically a pulp consistency of 8-18%) is connected to a tipping chute 2 for forwarding to an existing first MC mixer 3. The first mixer 3 is a so-called dynamic mixer, in which a motor-driven rotor agitates the pulp in at least one narrow fluidization  
25 gap. The dynamic mixer is preferably a mixer type which corresponds to that which is shown in US433920, in which a first cylindrical fluidization zone is formed between the rotor and the housing and a second fluidization zone is formed between a radially directed  
30 rotor part and housing, which mixer is hereby introduced as a reference. A mechanical agitation is required in order to obtain a uniform admixture of the chemical charge in question in the whole of the pulp suspension, with the aim of the pulp being  
35 bleached/treated uniformly throughout the whole of the volume of the pulp.

- 7 -

An admixture of chemicals, chiefly oxygen, takes place in the first MC mixer 3, after which the pulp was, in the existing system, fed to an oxygen reactor 6.

The combination of a first MC pump 1 followed closely by an MC mixer 3 can be termed a "perfect pair". This is the case since the pump primarily pressurizes the pulp flow to a given degree, thereby facilitating a finely divided supply of the oxygen to the MC mixer which follows directly thereafter.

10 In accordance with the invention, an upgrading of the oxygen delignification process is achieved by introducing a static mixer 8, i.e. a non-rotating or mechanically agitating mixer 8 for increasing the temperature by means of adding steam. The static mixer  
15 8 is preferably of a construction which has been shown in SE,C,512.192 (= PCT/SE00/00137), where steam is conducted in as thin jets through a number of holes which are uniformly distributed over the periphery of a pulp-conveying pipe, which mixer is hereby introduced  
20 as a reference.

The static mixer 8 is arranged directly after the oxygen reactor 6 and followed by a second MC pump 4 and a second agitating MC mixer 5, of the same type as the mixer 3, which acts directly after the MC pump 4. The  
25 system is assembled such that the coupling pipe 6 forms a first delignification zone between the outlet of the first MC mixer 3 and the inlet of the non-rotating mixer 8, which zone gives rise to a dwell time  $R_T$  of 2-20 minutes, preferably 2-10 minutes and even more  
30 advantageously 3-6 minutes.

The second MC pump 4 is controlled such that the resulting pressure in the dwell line 6 is preferably in the interval 0-6 bar, preferably 0-4 bar. Preferably, the second pump 4 is controlled by means of its  
35 rotational speed being controlled by a control system PC depending on the pressure which prevails, and is detected, in the first delignification zone 6.

- 8 -

The temperature in the whole of the first delignification zone 6 can be kept low, preferably at the level which the system allows without adding steam, but preferably with the pulp entering the first delignification zone being at a temperature of about 85°C,  $\pm 10^\circ\text{C}$ .

The non-rotating mixer 8 is connected in after the first delignification zone, as are then the second MC pump 4 followed by the second MC mixer 5. This second "perfect pair" combination is controlled such that the resulting pressure in the oxygen reactor 10, which forms a second delignification zone, reaches a level of at least 3 bars overpressure at the top of the reactor. In conventional applications, the pressure in the second MC mixer should be at least 4 bar higher than the pressure in the first MC mixer; alternatively, the increase in pressure in the second pump should reach 4 bar. In connection with practical implementation in conventional oxygen stages, an initial pressure is obtained within the interval 8-10 bar, corresponding to the pressure at the inlet to the reactor.

In accordance with the invention, the temperature of the pulp in the second delignification zone is increased by supplying steam to the non-rotating mixer directly after the first delignification zone and before the pressure-raising pump 4 comes into play. The steam supply is expediently controlled using a control system TC, which comprises a control valve V on the line 7 for the steam supply and a feeding-back measurement of the temperature of the pulp which is leaving the mixer. The temperature is expediently raised to a level of  $100^\circ\text{C} \pm 10^\circ\text{C}$ , but preferably at least  $5^\circ\text{C}$  higher than the temperature in the first delignification zone. As a result of the steam being added before the pulp is given the higher pressure



- 9 -

which is required for the final phase of the delignification:

- a higher temperature can be obtained
- the pressure of the available steam does not need to be so high
- the mixers for adding chemicals/admixing oxygen do not need to be burdened with a supply of steam as well, which will otherwise reduce their efficiency.

The volume of the second delignification zone, i.e. the second reactor, is expediently designed such that it is at least 10 times greater than the volume of the first delignification zone, i.e. a retention time of at least 20-200 minutes, preferably 20-100 minutes and even more advantageously within the range 50-90 minutes.

Figure 2 diagrammatically shows the kinetics of the oxygen delignification and the advantages with regard to the principles of kappa number reduction which are obtained relative to the prior art. Curve P1 shows the principle of a reaction course during the initial phase of the delignification. This part of the delignification proceeds relatively rapidly and is typically essentially complete after a good 20 minutes. However, after a relatively short time, typically only 5-10 minutes, the final phase P2 of the delignification takes over and begins to dominate as far as the resulting delignification of the pulp is concerned. A typical subdivision of the delignification into two stages in accordance with the prior art is shown at line A, with stage 1 being to the left of the line A and stage 2 being to the right of the line A. It follows from this that two different dominating processes, i.e. the initial phase of the delignification on the one hand, but also its final phase, actually take place in stage 1. It can be concluded from this that it becomes impossible to

- 10 -

optimize the process conditions in stage 1 for both these delignification phases.

Instead, a subdivision of the delignification into two stages in accordance with the invention is shown as a line B, a stage 1 is to the left of the line B and stage 2 is to the right of the line B. This makes it possible to optimize each stage for the process which dominates in the stage. The curve  $H_A$  shows the temperature integral plotted against time (the H factor) which is typically obtained when implementing a delignification process in two stages in accordance with the prior art, corresponding to the line A.

As can be seen from the figure, it is possible to use the stage subdivision in accordance with the invention to obtain an H factor which is higher than that which is typically obtained in current installations. This can be done without foregoing demands for high selectivity over the oxygen delignification system.

The invention also opens up ways of upgrading, with a small investment, an existing 1-stage process of comparatively low selectivity to a 2-stage system of better selectivity without having to build a new large reactor or even two such reactors. According to the invention, the initial phase of the oxygen delignification is dealt with in the prereactor, after which the temperature in the existing reactor can even be increased, if so required, in association with the conversion, and an increased H factor can in this way be combined with increased selectivity.

The invention can be modified in a number of ways within the context of the inventive concept. For example, the first delignification zone can consist of a "preretention tube" which is vertical but in which the pressure in some part of this "preretention tube", including its bottom, is at least 4 bar lower than the pressure in the initial part of the second delignification zone.

- 11 -

Further delignification zones, or intermediate washing/bleaching or extraction of the pulp, can be introduced between the first and second delignification zones according to the invention. For example, a third  
5 "perfect pair" combination, i.e. a pump with a mixer following it, can be arranged between the zones. What is essential is that the first delignification zone is characterized by a lower pressure, a short dwell time and a moderate temperature, and that the concluding,  
10 final delignification zone is characterized by a higher pressure (a pressure which is at least 4 bar higher than that of the first zone), a longer dwell time (a dwell time which is at least 10 times longer than that in the first zone) and an increased temperature (a  
15 temperature which is preferably at least 5 degrees higher than that in the first zone).

Where appropriate, it should be possible to charge a first mixer, or an intermediate mixer in a third  
20 "perfect pair" combination, with oxygen, at least some part of which is blown off from the reactor 10. The economic basis for such a recovery of oxygen is poor since the cost of oxygen is relatively low.

In order to ensure optimal process conditions, one or other, preferably the second, or both of the MC  
25 pumps can be rotation speed-controlled in dependence on the pressure in the first delignification zone.

The invention can also be modified by a number of varying additions of other chemicals either together with the oxygen or separately from the addition of  
30 oxygen, in a separate adding position, which chemicals are selected and suitable for the specific fibre line and the pulp quality in question, such as  
- alkali/NaOH for adjusting the pH level to that which is suitable for the pulp quality in question,  
35 - agents for protecting cellulose, for example  $MgSO_4$  or other alkaline earth metal ions or compounds thereof;

- 12 -

- additions of complexing agents which are performed prior to adding oxygen, with subsequent removal of precipitated metals, where appropriate,
  - chlorine dioxide;
  - 5 - hydrogen peroxide or organic or inorganic peracids or salts thereof;
  - free-radical capturing agents, such as alcohols, ketones, aldehydes or organic acids; and
  - carbon dioxide or other additives.
- 10           Where appropriate, it should also be possible to degas exhaust gases (residual gases) in immediate conjunction with the second pump, preferably by means of the pump being provided with internal degassing, preferably a pump termed a "degassing pump".